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6. AUTHOR(S) M. W. Barsoum and S. R. Kalidindi				
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13. ABSTRACT (Maximum 200 words) The goals of the proposed work were to elucidate the microstructural and atomic origins of the mechanical properties exhibited by Ti ₃ SiC ₂ . The focus is on the micro-mechanisms of inelastic deformation and their contribution to the damage tolerance exhibited by this compound. In addition to being technologically important, a basic study of the structure-property relations in this compound will be beneficial to understanding the behavior of the entire class of layered ternary carbides and nitrides. There are over 60 of these identified to date. During this work we conducted and successfully completed an extensive investigation of the mechanical response at two different length scales – macro (simple compression tests) and micro scale (nano-indentations). Additionally, we also completed extensive investigations on graphite. Our measurements revealed several unique features that are common to both of these layered materials, in comparison to other conventional ceramics. Our studies have revealed that the reversible, rate-independent, hysteretic stress-strain curves exhibited is attributable to incipient kink banding, which at higher stress levels (or temperatures) leads to irreversible deformation caused by formation of kink bands. Remarkably, the hysteretic energy dissipated in each cycle in the MAX phases can be as high as 25% of the elastic stored energy.				
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Final Report

Submitted to: Dr. David M. Stepp
U.S. Army Research Office
AMSRL-RO-PM (Materials Science Division)

Grant Title: Micromechanical Origins and Design Implications of Damage Tolerance in Ti_3SiC_2

Statement of the Problem Studied

In this project we systematically investigated the microstructural and atomic origins of the mechanical properties exhibited by Ti_3SiC_2 at two different length scale-macroscale and microscale at room and elevated temperatures. The focus was on the micro-mechanisms of inelastic deformation and their contribution to the damage tolerance exhibited by this promising compound. In addition to being technologically important, a basic study of the structure-property relations in this compound will be beneficial to understanding the behavior of the entire class of layered ternary carbides and nitrides. There are over 60 of these identified to date.

Summary of Our Most Notable Accomplishments

During the past 3 years we published 19 refereed papers [1-19] in some of the highest impact factor journals including: Nature Materials (1); Appl.Phys. Lett. (1), Carbon (1), Acta Mater. (4), J. Appl. Phys. (1), J. Mater. Res. (1), J. Electrochem. Soc. (4), J. Amer. Cer. Soc. (1), J. Alloys and Compds (3), and Scripta Mater. (1). As important the feature article and cover the July-August 2001 American Scientist was devoted to the MAX phases. We discovered a new physical phenomena – fully reversible plasticity - that resulted in a paper in Nature Materials [8]. We also explored the deformation of graphite and conclusively showed that it too deformed by the formation of kink bands. The importance of this discovery cannot be overemphasized because it finally explains the response of graphite to stress: a problem that until our work was not understood! Given the large number of papers published we cannot discuss all of our findings; instead we highlight our most important findings[‡].

1. Polycrystalline samples of Ti_3SiC_2 have been loaded cyclically in simple compression to stresses of about 1 GPa and strains of about 0.6% and they fully recovered upon the removal of the load, while dissipating about 25% of the mechanical energy. The stress-strain curves at room temperature outline fully reversible, reproducible, rate-independent, closed hysteresis loops that are strongly influenced by grain size, with the energy dissipated being significantly larger in the coarse grained material. At temperatures higher than 1000 °C, the stress-strain loops are open and the response becomes strain rate dependent. Cyclic hardening is observed at 1200 °C, for both fine and coarse-grained samples. This hitherto unreported phenomenon is attributed to the fully reversible formation and annihilation of incipient kink bands in room temperature deformation. At higher temperatures, the incipient kink bands dissociate and coalesce to form regular kink bands that are no longer reversible. (Ref. 8)

[‡] Please note it is possible to download most of the papers listed below in pdf format at <http://www.materials.drexel.edu/faculty/Barsoum/index.htm>.

2. We documented for the first time the load vs. depth-of-indentation response of Ti_3SiC_2 surfaces loaded with a 13.5 μm spherical tipped diamond indenter up to loads of 500 mN. Using orientation imaging microscopy (OIM), two groups of crystals were identified; one in which the basal planes were parallel to, and the other normal to, the surface. Not surprising for such a highly plastically anisotropic material, the response was anisotropic and comprised of two regimes. At higher loads (200-500 mN), delaminations occurred when loaded parallel to basal planes, and microcracking occurred when loaded normal to the basal planes. At lower loads (5 to 200 mN), the response was unique to Ti_3SiC_2 and resulted in almost fully reversible, closed hysteresis loops. Subsequent repeated (up to 5) loadings on the identical location not only resulted in fully reversible, reproducible hysteresis loops, but also caused a hardening of the indented volume. This response is attributed to the formation of incipient kink bands, followed by formation of regular kink bands. Remarkably, these dislocation-based mechanisms allow repeated loading of Ti_3SiC_2 without damage, while dissipating significant amounts of energy during each cycle. Also, the measurements of energy dissipated per cycle per unit volume from these experiments showed excellent agreement with the corresponding measurements from the simple compression tests described earlier, suggesting that the same underlying mechanisms continue to operate in this material even at the very high stress levels typical of the indentation experiments. (Ref. 1)
3. We documented the response of graphite single crystals to both a spherical and a Berkovich diamond indenter applied normal to the basal planes. The load vs. depth-of-indentation curves for the spherical indenter depended on load. At the lowest loads (≈ 5 mN), fully reversible hysteresis loops are observed. At intermediate loads (≈ 6 -200 mN) the first loops are almost fully reversible, and subsequent loops, in the same location, are not only fully reversible and reproducible, but also show hardening of graphite. At the highest loads (200–400 mN), significant (up to 60 μm) pop-ins are recorded, signifying delaminations, basal bond rupturing and massive damage. For the most part, no traces of the indentations made at loads up to 100 mN were found. In the 100-200 mN range, the indentations resulted in subtle crack patterns on the surface. At higher loads, the indentations lead to the formation of craters on the surface. The response to the Berkovich indenter is similar, except the various stages are shifted to lower loads. Since these observations are qualitatively almost identical to those for Ti_3SiC_2 - which is also a layered hexagonal material - they can be explained by the same mechanisms, namely, the formation of fully reversible incipient kink bands at the lower loads, and their dissociation into regular kink bands – that in turn leads to hardening - at higher loads. The presence of only basal slip, which precludes formation of dislocation entanglements, etc., results in the reversible to-and-fro motion of dislocations across considerable distances, which in turn results in the dissipation of significant amounts of energy during each cycle. Direct microstructural evidence was obtained for: i) the formation of kink bands; ii) the formation of a very large number of subgrains under the indenter and, iii) massive lattice rotations engendered by the formation of kink bands. These results very strongly suggest that kink bands play a much more important role in the deformation of graphite than had previously been reckoned. (Ref. 2)
4. The construction of a tensile deformation (“Ashby” type) map for Ti_3SiC_2 in the 1000 to 1200 $^\circ\text{C}$ for both fine (4 μm) and coarse (100 μm plate-like grains, 5 μm thick) grained samples of Ti_3SiC_2 . This entailed determining the constitutive equations for the tensile response for both microstructures as a function of strain rate. Over 200 individual tensile tests were carried out (Ref. 10)
5. Established that the strain rate exponent for both the fine and coarse-grained samples are quite high (≈ 0.5). At high strain rates, Ti_3SiC_2 is brittle; at lower strain rates considerable (up to 25% tensile strains at 1200 $^\circ\text{C}$ in air) deformation is possible. Since the strain rate exponent values are comparable to those of superplastic solids with grain sizes at least one order of magnitude smaller than the ones tested by us, the mechanism responsible for the strain rate sensitivity must be a new one. (Refs. 3, 10 and 14)
6. More interesting is the grain size exponent; the creep rate of Ti_3SiC_2 is a weak function of grain size (grain size exponent <1). This fact rules out diffusion and Coble creep. Of the remaining possible

mechanisms, the only ones that are consistent with our results are dislocation and grain boundary sliding. The evidence for dislocation creep despite exponents of < 3 is confirmed. More work is needed to conclusively prove grain boundary sliding. (Refs. 3, 10 and 14)

7. Determined conclusively that the mechanical response of Ti_3SiC_2 at temperatures close to the ductile-to-brittle transition (BDTT) ($\approx 1100^\circ\text{C}$) is dominated by stress relaxation processes; if the relaxation rate is rapid relative to the rate of accumulation of stresses the response is ductile, and vice-versa. (Refs. 3, 10 and 14)
8. Measured the times to failure and correlated them to the minimum creep rates via the Monkman Grant equation. (Ref. 10). We also showed that up to about 2 % strain to failure can be accommodated by plastic deformation alone. Above that, the strain is mostly due to cavitation and microcracking. The strains to failure at the low stresses and temperatures can exceed 8 % in tension. (Ref. 14)
9. Showed that the fracture toughness of Ti_3SiC_2 is quite high ($8\text{--}11 \text{ MPa}\sqrt{\text{m}}$) and rises with crack extension to $\approx 15 \text{ MPa}\sqrt{\text{m}}$. The latter is a record for a non-transforming ceramic. Most unexpectedly, but consistent with all other observations, the fracture toughness of Ti_3SiC_2 decreases above the DBTT. This is unique to the MAX phases and results from the nature of the DBTT. (Refs. 4 and 13).
10. The fatigue resistance of Ti_3SiC_2 is excellent and ceramic like in that the crack extension stress intensity plots are quite steep. The fatigue resistance is temperature independent up to 1100°C (Refs. 4 and 13).

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It is possible to download most of the papers listed below in pdf format at <http://www.materials.drexel.edu/faculty/Barsoum/index.htm>. Click on refereed publications; password is barsoum. **Highlighted papers noteworthy.**

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Awards:

1. First Place, American Ceramic Society Award for Student Poster Presentation Category in recognition of the poster presented at 2001 Conference on Advanced Ceramics and Composites, Cocoa Beach, FL.
2. M. W. Barsoum, Research Scholar Award: Drexel University, 2001.
3. S. R. Kalidindi, Research Achievement Award: Drexel University, 2003.

Conference Presentations and Posters

SMEC Conference, Florida International University, March 2003, Miami, FL.

1. "Nanoindentation of A Natural Nanolaminate: Ti_3SiC_2 ", M. W. **Barsoum**, A. Murugaiah, S. R. Kalidindi, T. Zhen and Y. Gogotsi. (INVITED)

Electrochemical Society Meeting, 203rd Meeting, Paris, France.

1. "Synthesis and Oxidation of Cr_2AlC and V_2AlC in Air", by S. Gupta and M. W. Barsoum,
2. "Oxidation of $\text{Ti}_{n+1}\text{AlX}_n$ where $n = 1-3$ and X is C and/or N", by **M. W. Barsoum**, N. Tzenov, A. Procopio, T. El-Raghy and M. Ali
3. "Long Time Oxidation Study Of Ti_3SiC_2 , $\text{Ti}_3\text{SiC}_2/\text{SiC}$ and $\text{Ti}_3\text{SiC}_2/\text{TiC}$ Composites in Air, by **M. W. Barsoum**, L. H. Ho-Duc, M. Radovic and T. El-Raghy

ACers, 105th Annual Meeting, Nashville, TN.

1. "Nanoindentation of a Natural Nanolaminate Material: Ti_3SiC_2 ", by **A. Murugaiah**, M. W. Barsoum, S. R. Kalidindi, T. Zhen and Y. Gogotsi
2. ACers, 105th Annual Meeting, Nashville, TN, "Nanolaminates, Kink Bands and Fully Reversible Dislocation-Based Deformation Up to 1 GPa in Ti_3SiC_2 ", by **T. Zhen**, M. W. Barsoum, S. Kalidindi, M. Radovic and A. Murugaiah
3. ACers, 105th Annual Meeting, Nashville, TN, "Synthesis and Oxidation Kinetics of Cr_2AlC in Air", by **S. Gupta** and M. W. Barsoum

MRS Meeting Spring 2003.

1. "Fully Reversible Dislocation-Based Deformation in a Nanolayered Carbide: Ti_3SiC_2 ", by M. W. Barsoum, T. Zhen, S. Kalidindi and A. Murugaiah.
2. MRS Meeting Spring 2003, "Deformation Processes During Nanoindentation of Ti_3SiC_2 ", Poster with A. Murugaiah, M. W. Barsoum, S. Kalidindi, and T. Zhen.

Annual International Conference on Advanced Ceramics & Composites; Cocoa Beach 2001 and 2002.

1. "Tensile Creep of Fine Grained Ti_3SiC_2 in the 1000-1200 °C Temperature Range", **M. Radovic**, M. W. Barsoum and T. El-Raghy and S. Wiederhorn. 2001
2. "Tensile Properties of Ti_3SiC_2 in the 25-1300°C Temperature Range", **M. Radovic**, M. W. Barsoum, T. El-Raghy, S. Wiederhorn. 2001
3. "Effect of Temperature, Strain Rate & Grain Size on the Mechanical Response of Ti_3SiC_2 in Tension", **M. Radovic**, T. El-Raghy, M. W. Barsoum & S. Wiederhorn 2001.

2000 and 2001 TMS Annual Meetings

1. " Deformation and Fracture of Ti_3SiC_2 During Creep in the 1000-1200°C Temperature Range". M. Radovic, M. W. Barsoum, T. El-Raghy and S. Wiederhorn.
2. "High Temperature Cyclic Fatigue Crack Growth in Monolithic Ti_3SiC_2 Ceramics", Proceeding of TMS Fall 2000 Meeting. K. Shirato, D. Chen, M. W. Barsoum, T. El-Raghy and R. O. Ritchie.

103rd Annual Meeting and Exposition of American Ceramic Soc. Indianapolis 2001

1. "Tensile Properties of Ti_3SiC_2 in the 25-1300°C Temperature Range", **M. Radovic**, M. W. Barsoum and T. El-Raghy.
2. "Deformation and Rupture of Ti_3SiC_2 During Creep in the 1000-1200°C Temperature Range", **M. Radovic**, M. W. Barsoum, T. El-Raghy and S. Wiederhorn.

2001 Solid State Studies in Ceramics, Gordon Conference, Aug. 2001- Posters

1. "Deformation and Rupture of Ti_3SiC_2 During Tensile Creep in the 1000-1200°C Temperature Range", with **M. Radovic**, T. El-Raghy and S. Wiederhorn.
2. "Ductile Machinable Carbides and Nitrides: A New Class of Solids", with **T. Zhen** and **A. Murugaiah**.

Oral Presentations:

Invited Presentations in which ARO funding was acknowledged. Unless otherwise noted all presentations were by M. W. Barsoum.

Rutgers University, New Brunswick, March 2003.
Oak Ridge national Laboratory, Oak Ridge, TN, Feb. 2003
ONERA, Paris, France, June 2002
University of Poitiers, France, June 2002
CNRS, Lyons, France, June, 2002.
GE Aircraft Engines, Cincinnati, OH May 2002.
U. of Missouri, Rolla, MO, April. 2002.
AIST, Sendai, Japan, March, 2002
Tohoku University, Sendai, Japan, March 2002
Cerratec Inc., Sendai, Japan, March, 2002
Drexel University, Phila., PA, Feb. 2002..
GE Aircraft Engines, Cincinnati, OH May 2002.
U. of Missouri, Rolla, MO, April 2002
AIST, Sendai, Japan, March, 2002
Tohoku University, Sendai, Japan, March 2002
Cerratec Inc., Sendai, Japan, March, 2002
Drexel U., Phila. PA, Feb. 2002.
U. of Pennsylvania, Philadelphia PA. Oct. 2001.
U. of Maryland, College Park, MD. Sept. 2001.
Naval Research Lab., Wash. DC., Sept. 2001.
Tech. Univ. Clausthal, Clausthal, Germany, June 2001.
U. of Hamburg, Hamburg, Germany, June 2001
Oak Ridge National Lab., June 2001. (Presented by M. Radovic)
U. of Ulm, Ulm, Germany, May 2001
AEA Technology, Oxford, England, April 2001.
Oxford University, Oxford, England, April 2001.
U. of Karlsruhe, Karlsruhe, Germany, March 2001.
Federal Instit. of Tech., Lausanne, Switzerland, Feb. 2001
Technical Univ. of Eindhoven, Holland, Feb. 2001
U. of Groningen, Holland, Feb. 2001
Max-Planck Institute, Stuttgart, Germany, Dec. 2000.
ABB Corp., Sweden, Dec. 2000.
Uppsala University, Sweden, Dec. 2000
Linköping University, Sweden, Dec. 2000.
Chalmers University of Tech., Sweden, Dec. 2000
Univ. of Vienna, Austria, Nov. 2000.
Ecole Centrale de Lyon, France, Nov. 2000.
Polytech. Instit. Milano, Italy, Nov. 2000

PI's and Graduate Students:

Michel Barsoum, PI.
Surya Kalidindi, Co-PI.
Tamer El-Raghy, Co-PI (no longer on the project).
Miladin Radovic, Ph.D., Graduated June 2001; now at ORNL
Anand Murugaiah, Ph.D. Candidate
T. Zhen, Ph.D. Candidate